

Statistical Method for the Determination of the Ignition Energy of Dust Cloud- Experimental Validation

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Abstract

Powdery materials such as metallic or polymer powders play a considerable role in many industrial processes. Their use requires the introduction of preventive safeguard to control the plants safety. The mitigation of an explosion hazard, according to the ATEX 137 Directive (1999/92/EU), requires, among other things, the assessment of the dust ignition sensitivity. PRISME laboratory (University of Orléans) has developed an experimental setup and methodology, using the Langlie test, for the quick determination of the explosion sensitivity of dusts. This method requires only 20 shots and ignition sensitivity is evaluated through the E_{50} (energy with an ignition probability of 0.5). A Hartmann tube, with a volume of 1.3 liters, was designed and built. Many results on the energy ignition thresholds of partially oxidised aluminium were obtained using this experimental device (Baudry, 2007) and compared to literature. E_{50} evolution is the same as MIE but their respective values are different and MIE is lower than E_{50} however the link between E_{50} and MIE has not been elucidated.

In this paper, the Langlie method is explained in detail for the determination of the parameters (mean value E_{50} and standard deviation σ) of the associated statistic law. The ignition probability versus applied energy is firstly measured for Lycopodium in order to validate the method. A comparison between the normal and the lognormal law was achieved and the best fit was obtained with the lognormal law .

In a second part, the Langlie test was performed on different dusts such as aluminium, cornstarch, lycopodium, coal, and PA12 in order to determine E_{50} and σ for each dust. The energies E_{05} and E_{10} corresponding respectively to an ignition probability of 0.05 and 0.1 are determined with the lognormal law and compared to MIE find in literature. E_{05} and E_{10} values of ignition energy were found to be very close and were in good agreement with MIE in the literature.

1 Introduction

Among the different accidental ignition sources, occurring in industrial situations, one of the most frequent is electrical discharge in the form of spark or arc. The sensitivity of dust to ignition by such sources can be measured in many ways. One of them, and probably the most used, is that of the Minimum Ignition Energy (MIE), defined as “the lowest electrical energy stored in a capacitor which upon discharge is just sufficient to affect ignition of the most ignitable mixture of given dust under specific test conditions” according to the EN 13821 standard (2002).

The European Standard (2002) recommends the method of MIE determination based on the classic Hartmann tube. The procedure is a rather complicated and consists in searching for "the highest value of energy E_1 at which ignition fails to occur in 10 successive attempts to ignite the dust–air mixture, and the lowest energy E_2 at which ignition occurs within up to 10 successive attempts". This implies that many attempts have to be carried out, and sometimes a considerable number of shots has to be made before the MIE value can be obtained.

Two kinds of apparatus are commonly used in regular laboratories to determine the MIE, namely:

- the Hartmann tube with a capacitor spark generator that is usually made by the laboratory itself according to the standard requirements and,
- the MIKE3 device, a commercially available apparatus described by Cesana and Siwek (2001). It uses two kinds of triggering circuits: the high voltage relay (Annex A2 in EN13821 standard) for low range MIE (1-3 mJ), and the electrode movement, using a two-electrode system (Annex A3 in EN13821 standard).

A. Janes et al (2008) compared the results obtained with two kinds of apparatus mentioned above and found that:

- the MIKE3 apparatus gives MIE values that are generally lower than those given by that of Hartmann. These differences are particularly emphasized for dusts with an MIE of between 1 and 10 mJ and over 100 mJ. These differences can alter the dust classification according to its sensitivity to electrostatic ignition sources
- Particle size distribution, dust concentration in the ignition area, the way of dust dispersion and delay between dust cloud generation and spark-over have a strong influence on the MIE.

These influences are reviewed in detail in the “Eckhoff book” (2005). In particular, the delay between dust cloud generation and ignition is very important - according to Eckhoff (2005) “the larger the delay, the lower the turbulence.” The relationship between MIE and the delay between dust dispersion and ignition is shown in Figure 1 (Eckhoff, 2005).

Figure 1

In another way, for the past few years the PRISME laboratory has been developing a statistical method –the Langlie method (1962)- originating in pyrotechnics to assess ignition sensitivity. In pyrotechnics this method, associated with a statistic law, is used to determine the ignition probability of a pyrotechnic material versus ignition energy, and gives the two parameters (the energy mean value, noted E_{50} , and the standard deviation σ) of the associated statistic law which is usually the normal law. Finally, this statistic law is used in two ways (see Figure 2): firstly to determine the ignition energy which ensures the running of the pyrotechnic system (noted E_{95} : ignition energy with a probability of 0.95) and secondly, for storage of the system in order to determine the higher energy which provides adequate safety (noted E_{05} : the ignition energy with a probability of 0.05). The Prisme laboratory is conducting a lot of research (Baudry 2007) on aluminum dust and assesses the sensitivity of dusts using the mean value E_{50} given by the Langlie method. The E_{50} measurement follows the same tendencies as the MIE, but it is not the MIE, and E_{50} is always higher than the MIE. The purpose of this paper is to link the MIE to an ignition probability. There are two major advantages in this: firstly the Langlie test gives the parameter of the statistics law after no more than 20 attempts and secondly, it is possible for the level of safety to be modulated by choosing the value of the probability (0.05, 0.1 or another one).

Figure 2

In this paper, the experimental device and the Langlie method are presented. The ignition probability of lycopodium dust is measured in order to evaluate two statistics law: normal and lognormal laws. Finally the Langlie test is performed on several dust and ignition energies with respectively an ignition probability of 0.05 and 0.1 and these are compared to the MIE found in the literature. The tested dusts are aluminium, cornstarch, lycopodium, coal, and PA12.

2 Experimental set-up

An experimental set-up was built in the laboratory in order to study the cloud ignition. It is directly inspired from the Hartmann tube. Efforts were focused in particular on:

- Low pressure injection of the system to reduce turbulence,
- The spark generator to control the applied energy,
- A test method to determine the MIE

This apparatus is presented in Figure 3, with the following main elements: a Hartmann tube, with an inner diameter of 70 mm and height of 330 mm, ($L/D \approx 4.7$) and Tungsten electrodes with a 2.4 mm diameter that are sharply pointed at an angle of 40° . This configuration provides the minimum erosion of the electrodes with the advantage of having a conical shape to generate the spark. The electrodes are installed at 110 mm from the bottom of the tube.

The spark gap is 3.5mm wide in this study. This distance provides the lowest energy according to the experiment carried out by Baudry (2007).

Figure 3

The power supply is designed to produce an arc at nearly constant power (voltage and current intensity are constant). Energy, in such a case, is only proportional to the duration of the spark. This time could be changed over the range of 1 μ s to 1 s. The proper, igniting arc is preceded by a pulse of high voltage helping to initiate the low voltage spark. The pulse shape is shown in Figure 3. The arc energy value achieved with such an arrangement is in the range from 10 mJ to 500 J, making it possible to measure the ignition energy of the less ignitable dusts.

The dust dispersion system creates a dust cloud by a blast of compressed air at a given pressure, from a tank through a valve with an opening time in the range of 60 ms (using electro valve technology) up to 1s. This delay is also adjusted to obtain a uniform dispersion of the dust in the tube. A lower overpressure (0.5 bar), in the compressed air tank, allows the turbulence to be reduced to below that associated with the 7 bars used for the MIKE3 apparatus, in accordance with the standard EN 13821 [1].

Figure 4a, shows a synopsis of the spark generator circuit and the arc voltage shape. A programmable logical controller pilots switches S1 and S2. Firstly, S1 is closed at t_0 , at the same time C is charged (this circuit is not represented), in a second phase, S2 is closed at t_1 and the discharge of C in the primary of high voltage self produces high voltage at its secondary. When the breakdown voltage of the air is achieved, at t_2 the voltage produced by the breakdown circuit is lower than that of the power generator so that the arc is automatically powered by the latter. The controller cuts power at t_3 . The value of the set point programmed is ($t_3 - t_2$) and is named: spark duration.

Figures 4b, 4c and 4d show the arc voltage and current intensity evolution for 3 programmed set points spark durations. The spark generator should allow us to program a spark duration equal to 0 μ s, but this set point always produces (due to the programmable controller technology) a first spark with an 8 μ s duration and the corresponding energy is equal to 9.44 mJ. This measurement is obtained by the integration of the power signal over time. This energy value is the minimum that can be produced with our spark generator.

Figure 4

The corresponding energies of sparks are given in Table 1.

Table 1

The delays between blast and spark generation can also be adjusted in the range of 0 to 1000 ms. In this study, in accordance with Eckhoff (2005), this delay is adjusted to 100 ms.

3 Test procedure

The determination of E_{50} is both prompt and reliable, so that its measurement is useful and often used in the field of safety on energetic materials. But E_{xx} energy is a statistical parameter which defines the evolution of the probability $xx\%$ versus ignition energy

E_{xx} . The main purpose of this work is to link the MIE to an ignition probability and discusses the choice of the statistic law between normal and lognormal law.

The test is divided into two parts. The first one uses a dichotomy approach to determine an approximation of the mean energy. After a suitable number of attempts, positive ignition occurs. The negative (no ignition) attempts are counted. Both are totalized and the mean energy approximation is given by:

$$E_{50} = \frac{X_m + X_M}{2} \quad (1)$$

where X_m represents the lowest value of the energy which gave a positive result (ignition) and X_M is the highest value which produced a failure. The standard deviation is given by:

$$\sigma_0 = N \frac{X_M - X_m}{8(n+2)} \quad (2)$$

where N is the total number of attempts and n the number of attempts between X_m and X_M .

The second part of this work relies upon a numerical treatment, with a normal law; an optimization of mean energy and standard deviation values are performed in order to improve agreement with the experiment. The opportunity to use a standard normal law or a lognormal law will be discussed in the experimental results. For the lognormal law the standard deviation parameter is calculated as:

$$\sigma_0 = N \frac{\ln(X_M) - \ln(X_m)}{8(n+2)} \quad (3)$$

where N , n , X_m and X_M are defined as previously.

The dusts reported in Table 2 were selected to measure ignition energy:

Table 2

The samples were dried at 70 °C for 24 hours, then screened by 80 and 50 µm sieves. In the case of coal dust, two size fractions were investigated: between 50 and 80 µm and below 50 µm.

The concentrations of dust samples, tested in the Hartmann tube, are given in Table 2.

Each type of dust was observed using a SEM microscope. SEM photography 2 shows a very smooth surface of the lycopodium spore which corresponds to the higher MIE of the different species of lycopodium. Figures 5 and 9 show that PA12 and aluminium particles are almost spherical. The two sorts of coal, in Figures 7 and 8, present a very

sharp shape. Finally, lycopodium and cornstarch are not spherical but their shape is very round (Figures 6 and 10).

4 Experimental results with Langlie test using the Normal Law

The Langlie method allows a correction of the mean value μ and the standard deviation σ with the normal law to fit the probability distribution of ignition. It can be observed that, generally, the corrected value of the average is near the initial value (E_{50}) but the standard deviation tends to increase as shown in Table 3.

Table 3

The increase of the standard deviation provides the enlargement of the distribution function therefore the ignition probability is sometimes different from 0 when energy is equal to 0. This problem appeared for Lycopodium ($P = 0.04$), Maïzena ($P = 10^{-6}$), Aluminium ($P = 10^{-4}$), PA12 ($P = 10^{-3}$), hard coal ($P = 10^{-3}$) and brown coal ($P = 0.2$) as shown in Figure 11. Another reason for this problem is the definition of the partition function of the normal law:

$$F(x) = \frac{1}{\sqrt{2\pi}\sigma} \int_{-\infty}^x \exp - \frac{(y - \mu)^2}{2\sigma^2} dy \quad (4)$$

In these conditions, the lower limit of the integral introduces probability unequal to zero for negative energies something which is without meaning.

Figure 11

For Lycopodium dust, considered as a reference, a statistical test was performed to determine the probability distribution of the ignition versus energy (see Figure 12). For each energy value, ten attempts were completed and positive shots were taken to calculate the ignition probability. Points 3 and 4 have probabilities which seem to be greater than we had hoped probably due to the sharpening of the electrodes.

Figure 12

The experimental data values are fitted with normal (eq.4) and lognormal laws which are given by:

$$F_{Log}(x) = \frac{1}{\sqrt{2\pi}\sigma} \int_0^x \frac{1}{y} \exp - \frac{(\ln(y) - \mu)^2}{2\sigma^2} dy \quad (5)$$

Mean values are in good agreement with experimental data, but it is difficult to obtain a good adjustment for a probability near 1 or 0. In this range, the best fit seems to occur for the lognormal law which gives the best agreement compared to the normal law as shown on Figure 6 and with the parameters of these laws as noted in Table 4. A standard deviation noted (*) is the standard deviation of X_i and the standard deviation (**) is the standard deviation for $\ln(X_i)$.

Table 4

The next part of this study consists of comparing the probability distribution obtained using the Langlie method in the cases of the normal law and the lognormal law. Two Langlie tests were carried out with lycopodium powder in order to determine the E_{50} energy and standard deviation (eq.1 to 3). Numerical treatment was applied and the ignition probability was plotted in the case of normal law and lognormal law with the corrected value of the mean and the standard deviation. For the Langlie test with normal law, numerical correction provides a very great standard deviation, while the value of the standard deviation for the lognormal method is close to the experimental values. In Figure 6 the ignition probability of lycopodium (test 1 & 2) is presented with a numerical correction of the Langlie method. In the case of the lognormal law, the Langlie method gives a better determination of the of the ignition probability as shown in Figure 12 compared to the ignition probability obtained with the normal law. The ignition probability is corrected to adjust to 0 when the energy is equal to 0. Thus it becomes possible to evaluate, in the case of the lognormal law fitting an MIE, an approximate value of between 20 and 23 mJ. The measured value is given by experimental data values near 19 mJ as shown on Figure 13.

Figure 13

In the last part of this study, several dusts were tested using the Langlie method and the lognormal law in order to determine the MIE values with a large energy range. The results of these flammability tests (according to the Langlie method - operating with the lognormal law) are summarized in Table 5.

Table 5

Figure 14 presents the ignition probability of the different dusts tested in our tube using the Langlie method and the lognormal law. The opportunity to evaluate a MIE appears clearly. It becomes possible to define the MIE as a probability of ignition. It is necessary to set a threshold that is representative of the explosion risk. For the tested dusts, this threshold had been set at 0.1 and 0.05. In the two cases the obtained values are very close except for brown coal of 50-80 μm where the relative difference is 16% as shown in Table 5. In these cases, the choice of the probability value (0.1 or 0.05) does not modify the MIE Values.

Figure 14

Table 6

The comparison of our results with the data in the literature is summarized in Table 7 for lycopodium, cornstarch, aluminium and coal dusts. The comparison of MIE, for lycopodium and cornstarch, can be made directly because the granulometry and composition of these mixtures do not change in great proportions. For aluminium powder, the granulometry is very large and modifies MIE significantly. For coal, dust discrepancies are important because compositions are not the same depending on the location of extraction.

For lycopodium, our measurement system is in accordance with the range of MIE given by MIKE3 apparatus but our value is greater than that given by the Hartmann tube apparatus from Janes (2008).

For cornstarch, good agreement is obtained with MIKE3 MIE but in this case the Hartmann MIE value is superior.

For Aluminium, our MIE measurement seems to be in agreement with the lower value given by MIKE 3 and lower than those obtained with the Hartmann tube. It can be noted that ranges of MIE with MIKE3 and Hartmann are very large. Moreover, the granulometry is not given in the work of A. James (2008).

Concerning coal dusts, the four different samples tested in our apparatus provide the MIE between 500 mJ and 25 J. The level of the MIE seems to be correct but due to the differences in composition and granulometry, it is very difficult to conclude with certainty.

Table 7

For Nylon, data values from Kwang Seok Choi (2005) were found in the literature. PA12 is a sort of Nylon, but two sorts of Nylon may probably exist. The Nylon powder tested has a 44 μm D_{50} but D_{50} of our particles is between 60 to 70 μm , that may explain why MIE value of our particles is superior to those obtained by Kwang Seok Choi (2005).

In the other cases it is interesting to note that the Langlie method - operating with a lognormal law - allows the determination of an E_{05} energy close to the MIE value measured using standard MIE tests. One main advantage of the Langlie method is the low number of shots necessary to obtain an MIE value (no more than twenty).

5 Conclusion

In this study, an original method for determination of the MIE has been presented. The experimental device, designed for the test, allows the ignition energy to be controlled by applying constant power. Firstly, the probability of ignition of a narrow size dispersive dust (Lycopodium) was measured. Experimental data values were best fitted by a lognormal law compared to the normal law which is traditionally used for the Langlie test.

In the second part of this study, the Langlie method was applied to Lycopodium and other dusts in order to determine the average and the standard deviation of each of them. These two parameters allowed us to calculate the energy (E_{05}) providing an ignition probability of 0.05 which is comparable to the MIE values measured with MIKE3 and Hartmann, applying standard MIE test EN 13821. This comparison shows a good accordance between the E_{05} and the MIE values. So the Langlie method - operating with lognormal law allows a significant reduction in the number of attempts needed (no more than 20) to determine E_{05} which can be representative of MIE.

The method and experimental set-up provide the possibility of measuring ignition energy over a very large range (0.01- 400 J). The calculation of the ignition probability for a given energy threshold allows the safety requirements to be adapted to the chosen risk level.

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Figure 1: Illustration of the influence of initial turbulence of explosive dust clouds on the minimum electric spark energies required for ignition. (copy of fig 1.40 from Eckhoff's book (page 39))

Figure 2: Ignition probability versus energy in Langlie test

Figure3: Experimental device, Hartmann tube and spark generator

Figure 4: Arc power, voltage and current intensity evolution versus time for 3 set points arc duration, a) "0" μ s, b) 20 μ s and c) 200 μ s

Figure 5: Ignition probability of some tested dust with the Langlie method in Hartmann tube

Figure 5: SEM photography of PA 12 particles

Figure 6: SEM photography of lycopodium spores

Figure 7: SEM photography of brown coal dust

Figure 8: SEM photography of hard coal dust

Figure 9: SEM photography of aluminium particles

Figure 10: SEM photography of cornstarch dust

Figure 11: Ignition probability of Lycopodium; experimental data's and fitted by normal and lognormal law

Figure 12: Representation of the ignition probability of the Lycopodium given by normal and lognormal law calculated with corrected values of the standard deviation and the average provided by the Langlie method.

Figure 13: Ignition probability of various dusts calculated with corrected values of the average and the standard deviation given by the Langlie method - operating with the lognormal law.

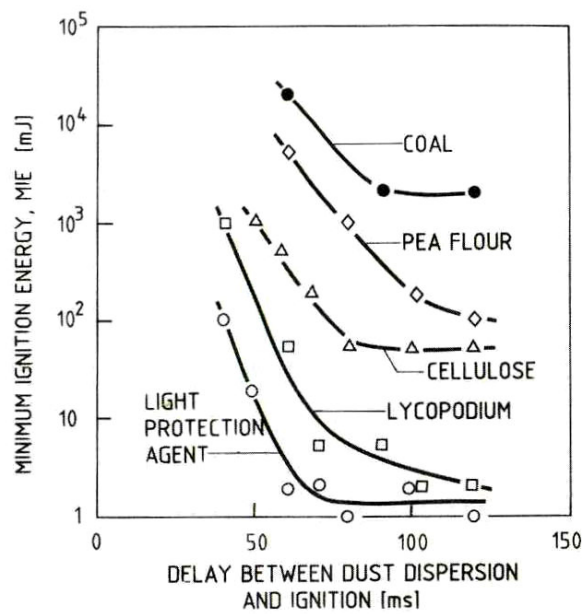


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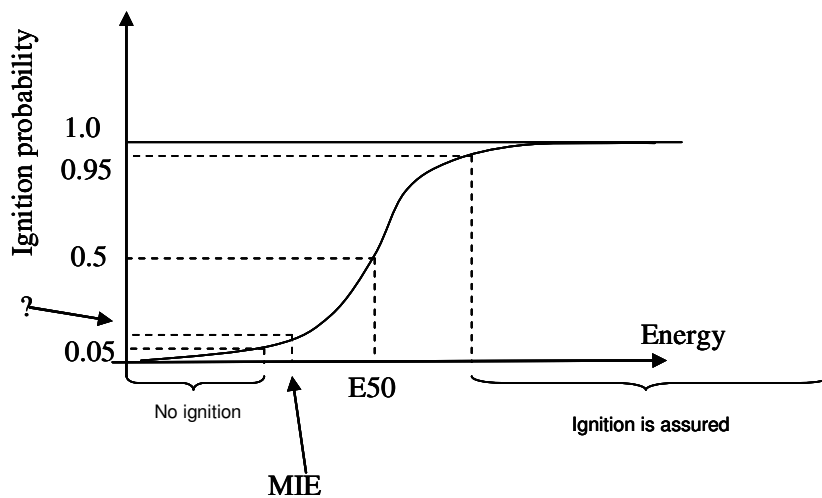


Figure 2: Ignition probability versus energy in Langlie test

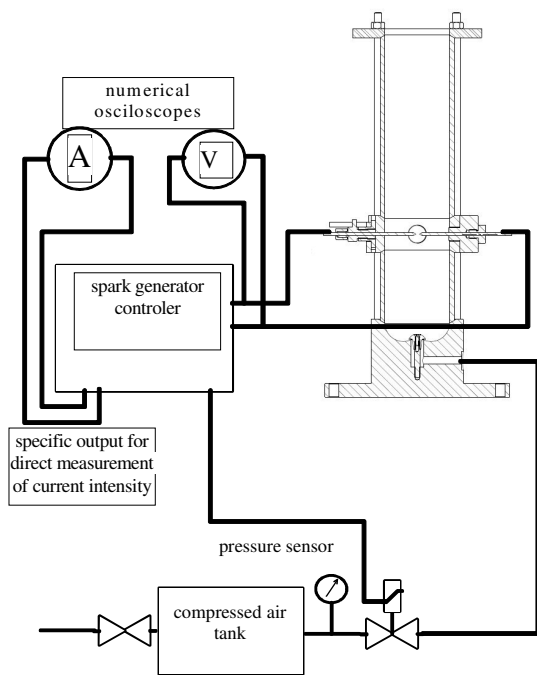
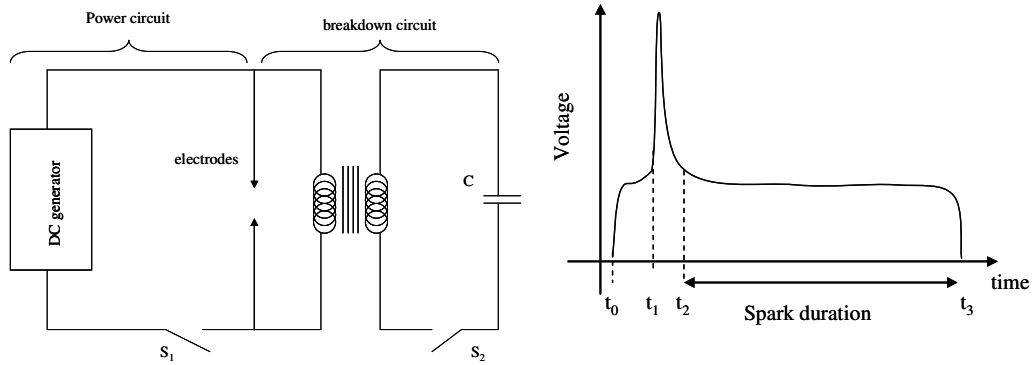
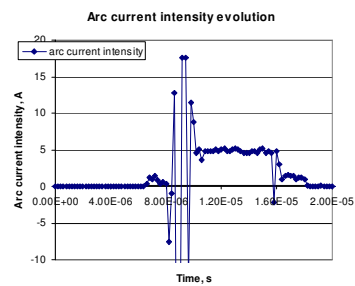
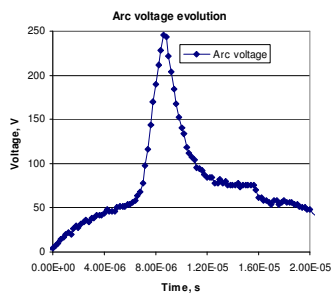


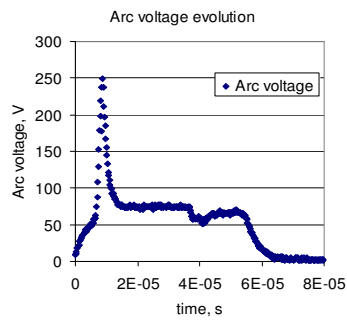
Figure 3: Experimental device, Hartmann tube and spark generator



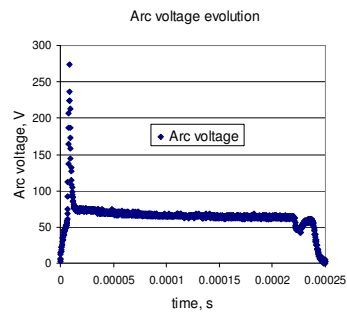
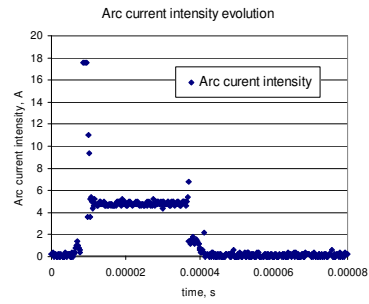
a)



b)



b)



c)

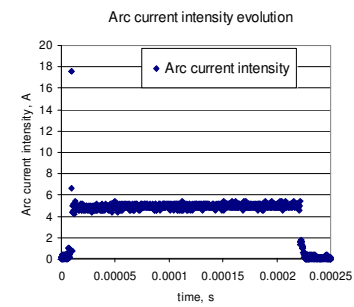


Figure 4: Synopsis of the spark generator circuit and arc voltage and current intensity evolution versus time for 3 set points arc duration, a) circuit and voltage shape, b) “0” μ s, c) 20 μ s and d) 200 μ s

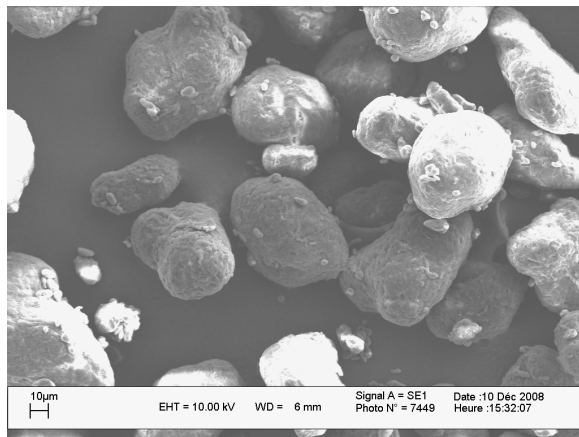


Figure 5: SEM photography of PA 12 particles

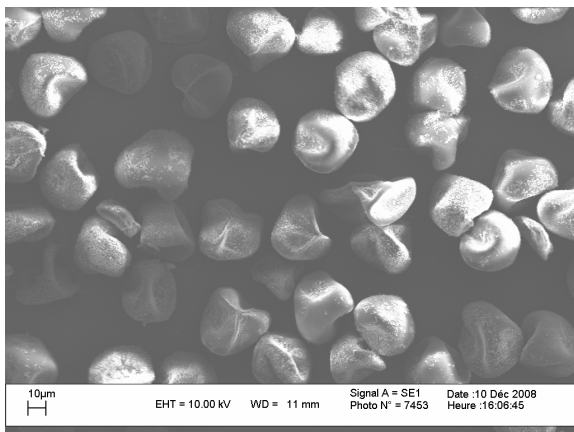


Figure 6: SEM photography of lycopodium spores

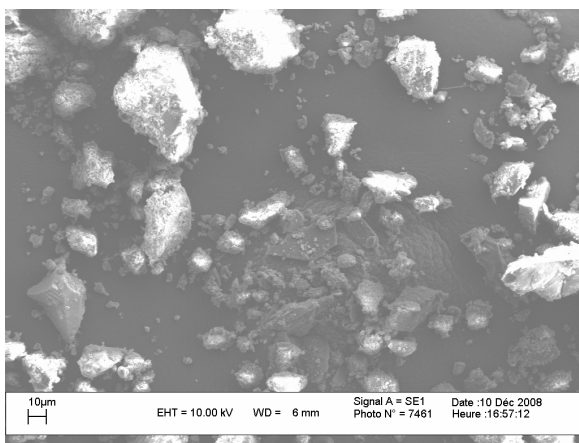


Figure 7: SEM photography of brown coal dust

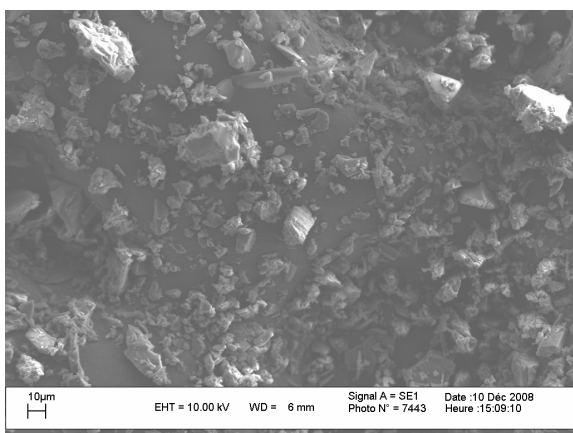


Figure 8: SEM photography of hard coal dust

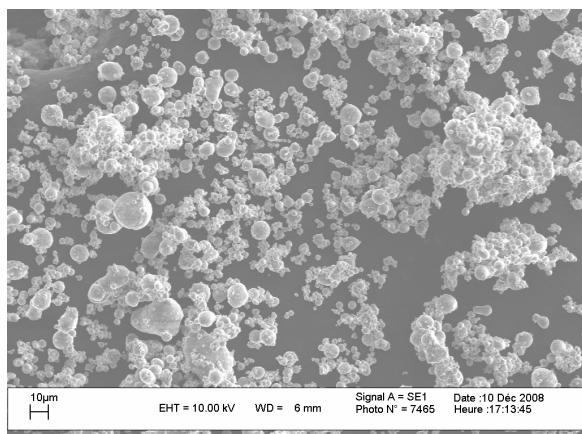


Figure 9: SEM photography of aluminium particles

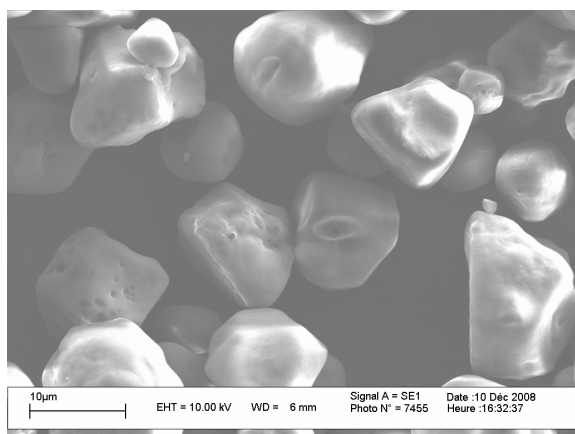


Figure 10: SEM photography of cornstarch dust

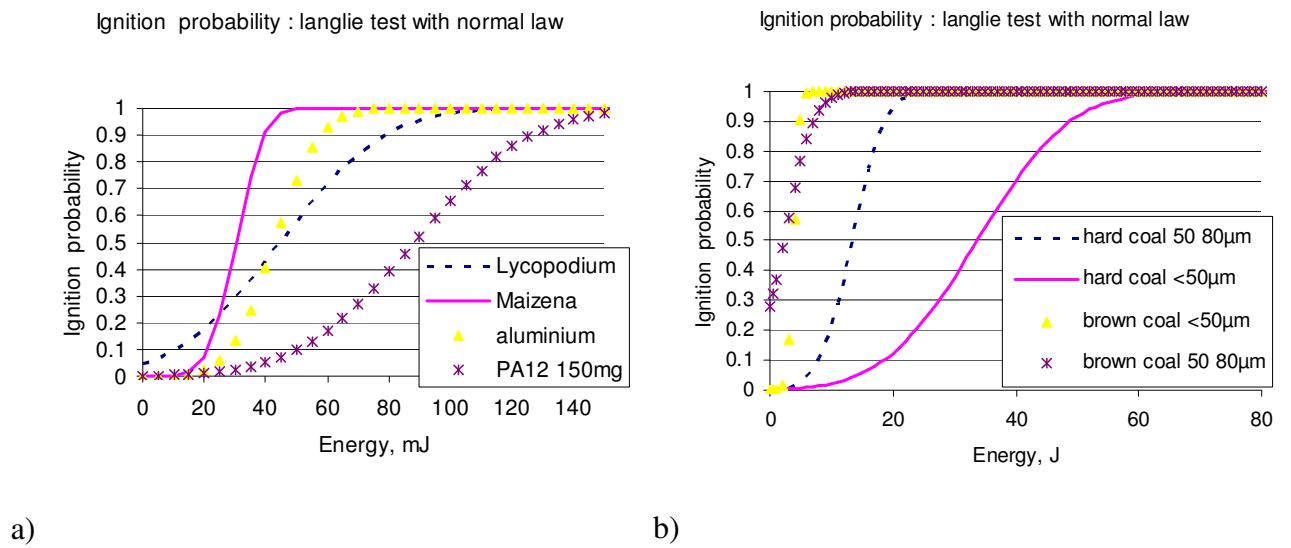


Figure 11: Ignition probability of dust tested using the Langlie method in the Hartmann tube

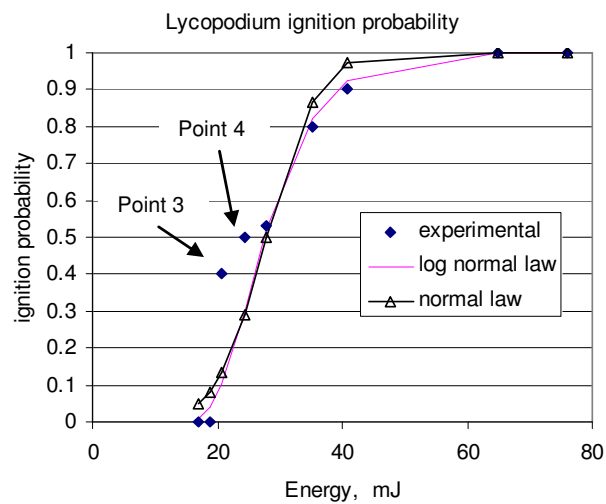


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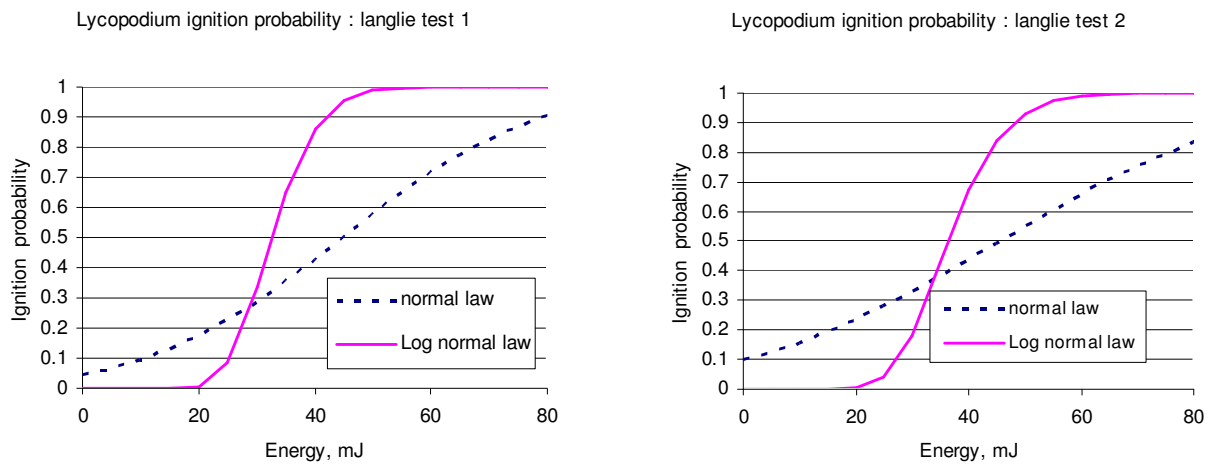


Figure 13: Representation of the ignition probability of the Lycopodium given by normal and lognormal law calculated with corrected values of the standard deviation and the average provided by the Langlie method.

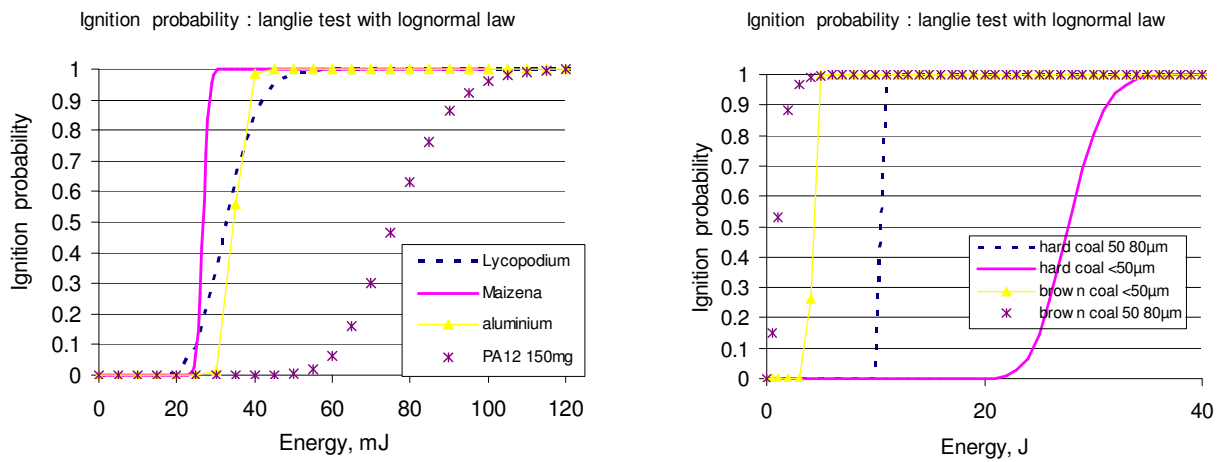


Figure 14: Ignition probability of various dusts calculated with corrected values of the average and the standard deviation given by the Langlie method - operating with the lognormal law.

Table 1: Energy value for given spark durations

Arc duration: set point, μs	Arc duration (measurement), μs	Arc energy, mJ
“0”	8	9.4
20	28	16.0
200	209	75.8

Table 2: Test conditions for different dusts

Dust	Mean diameter	Concentration mg/l
Lycopodium	31 μm monodispersive	160
cornstarch (Maizena)	$D_{50} = 12\mu\text{m}$	240
aluminium	$D_{50} = 27,5\mu\text{m}$	200
hard coal	50-80 μm	480
hard coal	<50 μm	480
brown coal	50-80 μm	480
brown coal	<50 μm	480
Polyamid PA 12	60 μm < D_{50} < 70 μm	80
Polyamid PA 12	60 μm < D_{50} < 70 μm	120
PA 12	60 μm < D_{50} < 70 μm	200

Table 3: Results of the Langlie test for given dusts

	experimental		Normal law	
	E50, mJ	σ	μ corrected, mJ	σ corrected
Lycopodium 1	47.63	9.49	44.95	26.55
Lycopodium 2	54.92	10.35	45.74	35.54
cornstarch	30.52	2.47	30.33	7.08
aluminium	46.97	5.45	42.91	11.56
PA12 100mg	121.13	9.51	118.10	19.34
PA12 150mg	87.29	11.07	88.33	29.53
PA12 250mg	69.09	1.03	67.83	2.67
	E50, J	σ	μ corrected, J	σ corrected
hard coal <50 μm	37.21	6.13	33.67	11.83
hard coal 50 80 μm	13.09	1.92	13.29	4.22
brown coal <50 μm	3.68	0.45	3.85	0.88
brown coal 50 80 μm	2.74	1.12	2.24	3.80

Table 4: Mean values and standard deviation for experimental data and fitted laws

	Mean value, mJ	Standard deviation	
experimental	27.5	9.8 (*)	0.263 (**)
Normal law	27.9	6.7	
Lognormal law	28.7	0.254	
Lycopodium test 1 Normal law	44.9	26.5	
Lycopodium test 1 Lognormal law	32.9	0.196	
Lycopodium test 2 Normal law	45.7	35.5	
Lycopodium test 2 Lognormal law	36.9	0.2133	

Table 5: Mean value and standard deviation obtained for different dusts with the Langlie method operating with the lognormal law.

	Lognormal Law	
	mean corrected, mJ	σ corrected
Liopodium 1	32.89	0.1916
Liopodium 2	37.22	0.2133
Maizena	26.79	0.0463
aluminium	34.73	0.0649
PA12 100mg	115.61	0.0978
PA12 150mg	76.91	0.1557
PA12 250mg	69.10	0.0186
hard coal <50 μ m	10.56 10^3	0.0140
hard coal 50 80 μ m	27.78 10^3	0.0952
brown coal <50 μ m	4.07 10^3	0.0257
brown coal 50 80 μ m	1155.52	0.6207

Table 6: Energy values obtained for two probabilities: 0.1 and 0.05 taken as the definition of the MIE

	MIE , mJ	
	probability = 0.1	probability = 0.05
Lycopodium 1	25.43	23.72
Lycopodium 2	27.68	25.61
Cornstarch (Maizena)	25.22	24.80
aluminium	31.89	31.15
PA12 100mg	101.51	97.97
PA12 150mg	62.24	58.82
PA12 250mg	67.46	67.01
hard coal <50µm	10.37 10 ³	10.32 10 ³
hard coal 50 80µm	24.48 10 ³	23.65 10 ³
brown coal <50µm	3.93 10 ³	3.90 10 ³
brown coal 50 80µm	430.21	343.36

Table 7: Comparison between our results and literature data values from Janes (2008)

Type of dust	Our apparatus	Mike 3	Hartmann tube
Lycopodium	26 mJ	10<MIE<30mJ	12<MIE<16
Cornstarch (maizena)	25 mJ	10<MIE<30mJ	45<MIE<58
Aluminium powder	32 mJ	30<MIE<100	45<MIE<58
Coal	Hard coal <50µm: 1037 Hard coal 50-80µm: 24 480 Brown coal <50µm: 3930 Brown coal 50-80µm: 430	1200 mJ<MIE	1000 mJ<MIE
Nylon and PA12	60 to 65 for PA12	35 mJ for Nylon	/